

12 Experimental techniques and chemical analysis

12.1 Experimental design

1 Name appropriate apparatus for the measurement of time, temperature, mass and volume, including:

(a) stop-watches

(b) thermometers

(c) balances

(d) burettes

(e) volumetric pipettes

(f) measuring cylinders

(g) gas syringes

2 Suggest advantages and disadvantages of experimental methods and apparatus

Instrument	Advantages	Disadvantages
Pipette	- Very high accuracy for small volumes - Easy to handle for fixed volumes - Ideal for preparing precise solutions - suitable for titrations	- Limited to small volumes - Time-consuming for multiple transfers - Requires careful technique to avoid error
Measuring Cylinder	- Can measure a wide range of volumes - Quick and simple to use - Inexpensive and widely available	- Less accurate than pipettes and burettes - Meniscus reading errors common - Not suitable for titrations

Burette	- Very precise for variable volumes - suitable for titrations - Allows controlled release of liquid	- More expensive and fragile - Requires skill to use correctly - Setup and cleaning take longer
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3 Describe a:

- (a) solvent as a substance that dissolves a solute
- (b) solute as a substance that is dissolved in a solvent
- (c) solution as a mixture of one or more solutes dissolved in a solvent
- (d) saturated solution as a solution containing the maximum concentration of a solute dissolved in the solvent at a specified temperature
- (e) residue as a substance that remains after evaporation, distillation, filtration or any similar process
- (f) filtrate as a liquid or solution that has passed through a filter

12.2 Acid-base titrations

1 Describe an acid-base titration to include the use of a:

- (a) burette
- (b) volumetric pipette
- (c) suitable indicator

AND 2 Describe how to identify the end-point of a titration using an indicator

- Step 1: add 25cm^3 of acid into a pipette after rinsing the pipette with acid and distilled water. Add this to a conical flask along with 3-5 drops of methyl orange indicator
- Step 2: Fill a burette with 50cm^3 of base add slowly add the base into the flask. After every addition, swirl the mixture.
- Step 3: once the solution changes colour permanently, the endpoint has been reached. Calculate the amount of base added(the amount needed to neutralise all the acid).
- Step 4: repeat the experiment multiple times and get the mean volume of base needed to neutralise the all the acid.

12.3 Chromatography

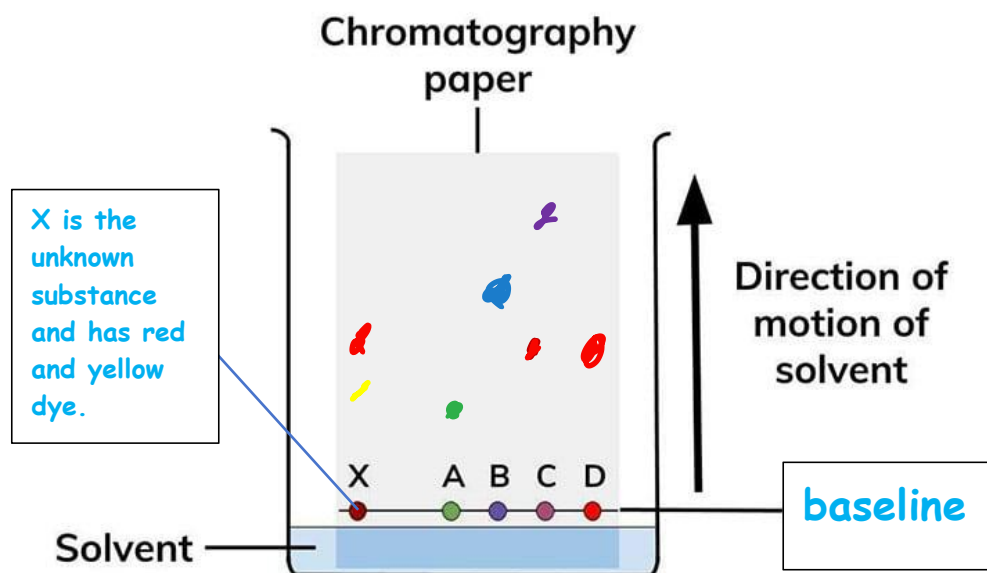
1 Describe how paper chromatography is used to separate mixtures of soluble coloured substances, using a suitable solvent

- Step 1: draw a pencil line(base line) on a chromatogram and put a drop of solution on the line and leave it to dry.
- Step 2: stand the chromatogram in a suitable solvent with the solvent level below the base line.
- The solvent rises and dissolves the drop, the mixture then separates into different substances(represented by different spots).
- Step 4: once the solvent level rises up most of the chromatogram, remove it and mark the solvent front.

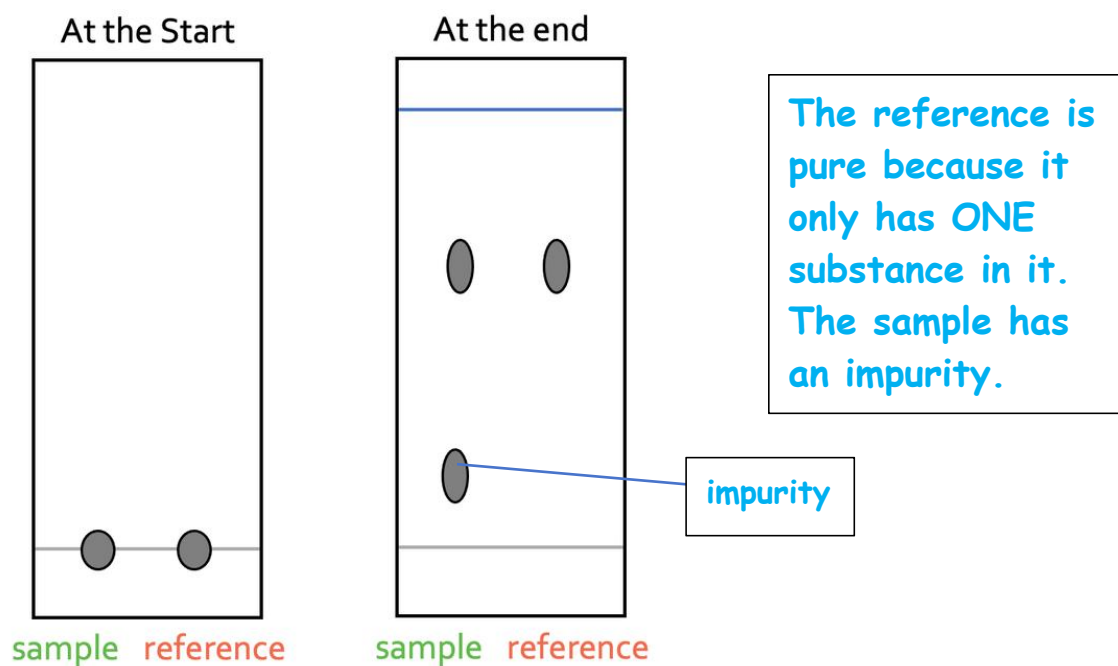
- Step 5: calculate the R_f .

2 Interpret simple chromatograms to identify:

(a) unknown substances by comparison with known substances



(b) pure and impure substances



3 Describe how paper chromatography is used to separate mixtures of soluble colourless substances, using a suitable

solvent and a locating agent **Knowledge of specific locating agents is not required**

- Perform the same steps, BUT apply a locating agent to make invisible spots visible.

4 State and use the equation for R_f :

$$R_f = \frac{\text{distance travelled by substance}}{\text{distance travelled by solvent}}$$

12.4 Separation and purification

1 Describe and explain methods of separation and purification using:

(a) a suitable solvent

- Example: To a mixture of sand and salt, add water. Salt dissolves in water but sand does not. Filter off the sand.

(b) Filtration

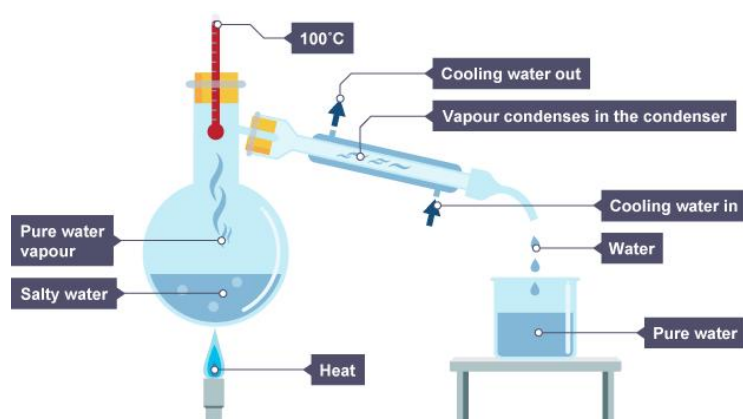
- The residue is the excess reactant/insoluble substance that gets collected on filter paper.
- The filtrate is the product that gets collected in the conical flask.



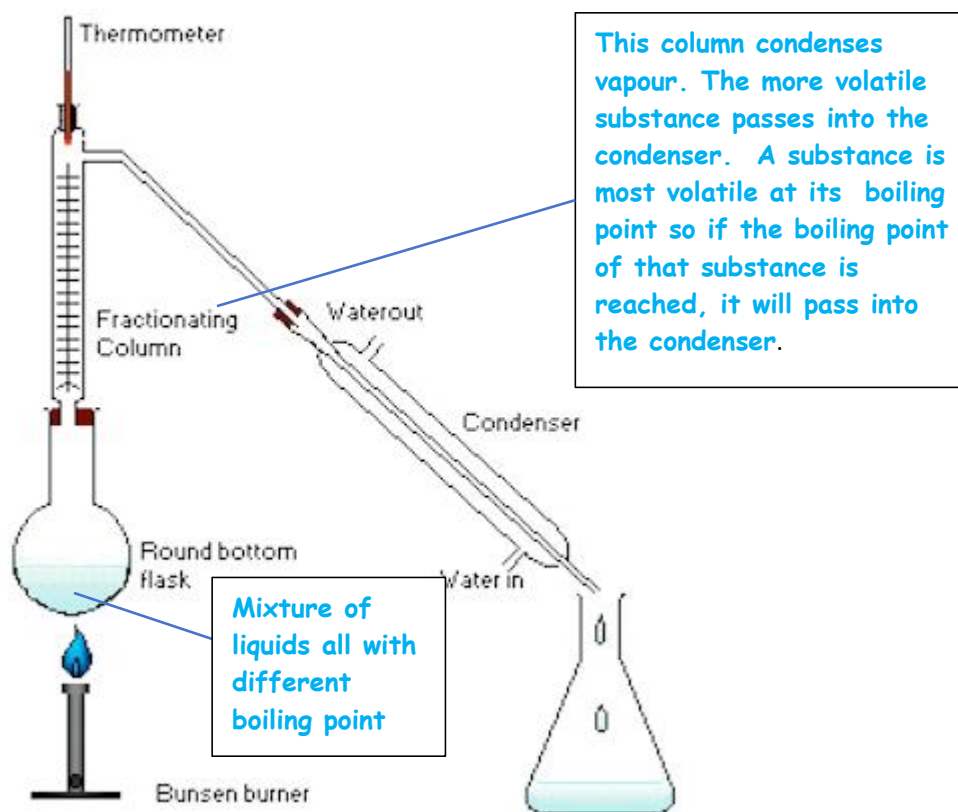
(c) crystallisation



(d) simple distillation



(e) fractional distillation



2 Suggest suitable separation and purification techniques, given information about the substances involved

- Add a suitable solvent to separate two or more solids mixed together.
- Crystallisation separates a soluble substance from its solution(in crystal form).
- Filtration separates an insoluble substance from a solution.
- Simple distillation separates soluble solid from its solution
- Fractional distillation separates a mixture of liquids.

3 Identify substances and assess their purity using melting point and boiling point information

- Melting point decreases if the substance has impurities. Boiling point increases if the substance has impurities.

12.5 Identification of ions and gases

1 Describe tests to identify the anions: [ppt.= preprecipitate]

(a) carbonate, CO_3^{2-} , by reaction with dilute acid and then testing for carbon dioxide gas

(b) chloride, Cl^- , bromide, Br^- , and iodide, I^- , by acidifying with dilute nitric acid then adding aqueous silver nitrate

(c) nitrate, NO_3^- , reduction with aluminium foil and aqueous sodium hydroxide and then testing for ammonia gas

(d) sulfate, SO_4^{2-} , by acidifying with dilute nitric acid and then adding aqueous barium nitrate

(e) sulfite, SO_3^{2-} , by reaction with acidified aqueous potassium manganate(VII)

anion	test	test result
carbonate, CO_3^{2-}	add dilute acid, then test for carbon dioxide gas	effervescence, carbon dioxide produced
chloride, Cl^- [in solution]	acidify with dilute nitric acid, then add aqueous silver nitrate	white ppt.
bromide, Br^- [in solution]	acidify with dilute nitric acid, then add aqueous silver nitrate	cream ppt.
iodide, I^- [in solution]	acidify with dilute nitric acid, then add aqueous silver nitrate	yellow ppt.
nitrate, NO_3^- [in solution]	add aqueous sodium hydroxide, then aluminium foil; warm carefully	ammonia produced
sulfate, SO_4^{2-} [in solution]	acidify with dilute nitric acid, then add aqueous barium nitrate	white ppt.
sulfite, SO_3^{2-}	add a small volume of acidified aqueous potassium manganate(VII)	the acidified aqueous potassium manganate(VII) changes colour from purple to colourless

2 Describe tests using aqueous sodium hydroxide and aqueous ammonia to identify the aqueous cations:

(a) aluminium, Al^{3+}

(b) ammonium, NH_4^+

(c) calcium, Ca^{2+}

(d) chromium(III), Cr^{3+}

(e) copper(II), Cu^{2+}

(f) iron(II), Fe^{2+}

(g) iron(III), Fe^{3+}

(h) zinc, Zn^{2+}

cation	effect of aqueous sodium hydroxide	effect of aqueous ammonia
aluminium, Al^{3+}	white ppt., soluble in excess, giving a colourless solution	white ppt., insoluble in excess
ammonium, NH_4^+	ammonia produced on warming	—
calcium, Ca^{2+}	white ppt., insoluble in excess	no ppt. or very slight white ppt.
chromium(III), Cr^{3+}	green ppt., soluble in excess	green ppt., insoluble in excess
copper(II), Cu^{2+}	light blue ppt., insoluble in excess	light blue ppt., soluble in excess, giving a dark blue solution
iron(II), Fe^{2+}	green ppt., insoluble in excess, ppt. turns brown near surface on standing	green ppt., insoluble in excess, ppt. turns brown near surface on standing
iron(III), Fe^{3+}	red-brown ppt., insoluble in excess	red-brown ppt., insoluble in excess
zinc, Zn^{2+}	white ppt., soluble in excess, giving a colourless solution	white ppt., soluble in excess, giving a colourless solution

3 Describe tests to identify the gases:

(a) ammonia, NH_3 , using damp red litmus paper

(b) carbon dioxide, CO_2 , using limewater

(c) chlorine, Cl_2 , using damp litmus paper

(d) hydrogen, H_2 , using a lighted splint

(e) oxygen, O_2 , using a glowing splint

(f) sulfur dioxide, SO_2 , using acidified aqueous potassium manganate(VII)

gas	test and test result
ammonia, NH_3	turns damp red litmus paper blue
carbon dioxide, CO_2	turns limewater milky
chlorine, Cl_2	bleaches damp litmus paper
hydrogen, H_2	'pops' with a lighted splint
oxygen, O_2	relights a glowing splint
sulfur dioxide, SO_2	turns acidified aqueous potassium manganate(VII) from purple to colourless

4 Describe the use of a flame test to identify the cations:

(a) lithium, Li^+

(b) sodium, Na^+

(c) potassium, K^+

(d) calcium, Ca^{2+}

(e) barium, Ba^{2+}

(f) copper(II), Cu^{2+}

metal ion	flame colour
lithium, Li^+	red
sodium, Na^+	yellow
potassium, K^+	lilac
calcium, Ca^{2+}	orange-red
barium, Ba^{2+}	light green
copper(II), Cu^{2+}	blue-green

END OF CHAPTER

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